

This Page Is Inserted by IFW Operations  
and is not a part of the Official Record

## **BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning documents *will not* correct images,  
please do not report the images to the  
Image Problem Mailbox.**

THIS PAGE BLANK (USPTO)

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
18 October 2001 (18.10.2001)

PCT

(10) International Publication Number  
**WO 01/77279 A1**

(51) International Patent Classification<sup>7</sup>: **C11D 17/00**,  
3/20, 3/00, 1/94, 3/48

(21) International Application Number: PCT/US01/10688

(22) International Filing Date: 3 April 2001 (03.04.2001)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:  
09/543,917 6 April 2000 (06.04.2000) US

(71) Applicant: **COLGATE-PALMOLIVE COMPANY**  
[US/US]; 300 Park Avenue, New York, NY 10022 (US).

(72) Inventors: **LEONARD, Isabelle**; 25, Vieille Voie de Ton-  
gres, B-4451 Voroux-Lez-Liers (BE). **DORMAL, Didier**;  
Deigne 111, B-4920 Aywaille (BE).

(74) Agent: **NANFELDT, Richard, E.**; Colgate-Palmolive  
Company, 909 River Road, P.O. Box 1343, Piscataway, NJ  
08855-1343 (US).

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU,  
AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU,  
CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM,  
HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK,  
LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX,  
MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL,  
TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW.

(84) Designated States (*regional*): ARIPO patent (GH, GM,  
KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian  
patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European  
patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE,  
IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF,  
CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

**Published:**

- with international search report
- before the expiration of the time limit for amending the  
claims and to be republished in the event of receipt of  
amendments

For two-letter codes and other abbreviations, refer to the "Guid-  
ance Notes on Codes and Abbreviations" appearing at the begin-  
ning of each regular issue of the PCT Gazette.

(54) Title: ACIDIC CLEANING COMPOSITION

(57) Abstract: An improvement is described in microemulsion compositions which are especially effective in disinfecting the sur-  
face being cleaned and in the removal of oily and greasy soil without leaving streaks which contains a mixture of nonionic surfactants,  
an anionic surfactant, an amphoteric surfactant, a disinfectant agent, a proton donating agent, a water soluble solvent, and water.

WO 01/77279 A1

## ACIDIC CLEANING COMPOSITION

Field of the Invention

This invention relates to an improved multi purpose liquid cleaner in a  
5 microemulsion form, to be used neat, in particular for cleaning and disinfecting hard  
surfaces and which is effective in sanitizing surfaces, in removing grease soil and also  
dries fast leaving the surfaces streak free.

Background of the Invention

Disinfectant composition based on cationic and nonionic are well known.  
10 However, these compositions while very efficient in disinfecting surfaces, generally do  
not remove grease and oil as desired; hence, leaving residues and streaks on surfaces.  
Addition of an efficient anionic surfactant cleaner, to the cationic surfactant, either  
creates instability problems or deactivates the disinfectant behavior of the cationic.  
Moreover, most cationic compositions are not environmentally friendly. Anionic and  
15 nonionic mixtures have a good grease removal properties, but do not perform at all to  
sanitize the surface being cleaned.

Summary of the Invention

The instant compositions are cationic free and exhibit good grease removal  
properties combined with excellent disinfecting properties and the compositions do not  
20 leave streak or residue on the surface being cleaned.

The described compositions are to be used mainly neat in a spray form. The  
compositions contain a lower level of surfactant than current all purpose cleaning  
compositions and have a richer level of solvent than surfactant. In order to have a  
product which leaves the surfaces shiny after cleaning, the instant compositions are  
25 builder free.

A safe solvent system used in the instant compositions which has been found  
effective are glycol ethers such as propylene glycol butyl ether (PNB) alone or in  
combination with ethanol. PNB brings a significant benefit in degreasing performance  
and also promotes a fast drying out of the surface which has been cleaned. Oily

material such as essential oil or perfume, when added to a composition, usually increase the streaks on surfaces.

The combination of a glycol ether with the surfactant system of the present invention allows a faster water film dry out time than obtained with current commercial spray formulations. It is important to select the perfume such as an essential oil not only to satisfy the cosmetic and the marketing needs, but also to have a minimum of residue left on the surface being cleaned. The selection of essential oils is also important because not only they will communicate the efficacy of the product to the consumer but also because they are effective bactericide.

In one aspect, the invention generally provides a stable, clear multi purpose, hard surface cleaning composition especially effective in disinfecting the surface being cleaned and in the removal of oily and greasy oil while a fast dry out time and without leaving streaks on the surface being cleaned. The compositions include approximately, on a weight basis:

from 0 to 10% of a nonionic surfactant formed from a C<sub>8</sub>-C<sub>18</sub> alkanol and about 6 to about 9, preferably about 7 to about 8.5 moles of ethylene oxide;

0.1% to 4% of an ethoxylated nonionic surfactant formed from a C<sub>9</sub>-C<sub>11</sub> alkanol and about 2 to about 3 moles of ethylene oxide;

0.1% to 10% of an amphoteric surfactant;

from 0 to 5%, more preferably 0.1 to 3% of an anionic surfactant

from 0 to 4%, more preferably 0.1% to 1% of at least one disinfecting agent such as essential oil or perfume;

from 0.1 to 10% of at least one amphoteric surfactant;

0.1% to 5%, more preferably 0.3% to 3% of a proton donating agent;

0 to 6% of at least a water soluble solvent which is a C<sub>1</sub>-C<sub>4</sub> alkanol;

0 to 1.5 %, more preferably 0.3% to 1.5% of triethanol amine;

1% to 10% of a blend of water soluble glycol ethers; and

the balance being water.

Detailed Description of the Invention

The present invention relates to a stable hard surface cleaning composition approximately by weight:

0% to 10% of an ethoxylated nonionic surfactant formed from a C<sub>8</sub>-C<sub>18</sub> alkanol

5 and about 6 to about 9, preferably about 7 to about 8.5 moles of ethylene oxide;

0.1% to 4% of an ethoxylated nonionic surfactant formed from a C<sub>9</sub>-C<sub>11</sub> alkanol and about 2 to about 3 moles of ethylene oxide;

0 to 5%, more preferably 0.1 to 3% of an anionic surfactant;

10 0 to 4 %, more preferably 0.1% to 1% of at least one disinfecting agent such as a essential oil or perfume,

0.1% to 10% of an amphoteric surfactant,

0.5% to 5% of a propylene glycol alkyl ether, wherein the alkyl group has 1 to 5 carbon atoms, such as propylene glycol N-butyl ether;

15 0.5% to 5% of an ethylene glycol alkyl ether, wherein the alkyl group has 1 to 5 carbon atoms or a diethylene glycol alkyl ether, wherein the alkyl group has 1 to 5 carbon atoms such as diethylene N-butyl ether;

0 to 6%, more preferably 0.1% to 5% of a C<sub>1</sub>-C<sub>4</sub> alkanol such as ethanol;

20 0.1% to 5%, more preferably 0.3% to 3% of a proton donating agent selected from the group consisting of hydroxy containing organic acids such as lactic acid, citric acid or ortho hydroxy benzoic acid and inorganic acids such as a sulfuric acid or hydrochloric acid and mixtures thereof;

0 to 1.5%, more preferably 0.3% to 1.5% of triethanol amine; and

25 the balance being water, wherein the composition has a pH of 2.7 to 4.5, more preferably 3 to 4 and the composition does not contain an amine oxide surfactant, a cationic surfactant or an alkyl polyglucoside surfactant.

The disinfecting agent is selected from the group consisting of triclosan, perfumes, essential oils and mixtures thereof.

As used herein and in the appended claims the term "perfume" is used in its ordinary sense to refer to and include any non-water soluble fragrant substance or

mixture of substances including natural (i.e., obtained by extraction of flower, herb, blossom or plant), artificial (i.e., mixture of natural oils or oil constituents) and synthetically produced substance) odoriferous substances. Typically, perfumes are complex mixtures of blends of various organic compounds such as alcohols, aldehydes, ethers, aromatic compounds and varying amounts of essential oils (e.g., terpenes) such as from 0% to 80%, usually from 10% to 70% by weight, the essential oils themselves being volatile odoriferous compounds and also serving to dissolve the other components of the perfume.

Suitable essential oils are selected from the group consisting of: Anethole 20/21 natural, Aniseed oil china star, Aniseed oil globe brand,, Balsam (Peru), Basil oil (India), Black pepper oil, Black pepper oleoresin 40/20, Bois de Rose (Brazil) FOB, Borneol Flakes (China), Camphor oil, White, Camphor powder synthetic technical, Cananga oil (Java), Cardamom oil, Cassia oil (China), Cedarwood oil (China) BP, Cinnamon bark oil, Cinnamon leaf oil, Citronella oil, Clove bud oil, Clove leaf, Coriander (Russia), Coumarin 69°C (China), Cyclamen Aldehyde, Diphenyl oxide, Ethyl vanillin, Eucalyptol, Eucalyptus oil, Eucalyptus citriodora, Fennel oil, Geranium oil, Ginger oil, Ginger oleoresin (India), White grapefruit oil, Guaiacwood oil, Gurjun balsam, Heliotropin, Isobornyl acetate, Isolongifolene, Juniper berry oil, L-methyl acetate, Lavender oil, Lemon oil, Lemongrass oil, Lime oil distilled, Litsea Cubeba oil, Longifolene, Menthol crystals, Methyl cedryl ketone, Methyl chavicol, Methyl salicylate, Musk ambrette, Musk ketone, Musk xylol, Nutmeg oil, Orange oil, Patchouli oil, Peppermint oil, Phenyl ethyl alcohol, Pimento berry oil, Pimento leaf oil, Rosalin, Sandalwood oil, Sandenol, Sage oil, Clary sage, Sassafras oil, Spearmint oil, Spike lavender, Tagetes, Tea tree oil, Vanillin, Vetyver oil (Java), Wintergreen, Allocimene, Arbanex™, Arbanol®, Bergamot oils, Camphene, Alpha-Campholenic aldehyde, l-Carvone, Cineoles, Citral, Citronellol Terpenes, Alpha-Citronellol, Citronellyl Acetate, Citronellyl Nitrile, Para-Cymene, Dihydroanethole, Dihydrocarveol, d-Dihydrocarvone, Dihydrolinalool, Dihydromyrcene, Dihydromyrcenol, Dihydromyrcenyl Acetate, Dihydroterpineol, Dimethyloctanal, Dimethyloctanol, Dimethyloctanyl Acetate, Estragole, Ethyl-2 Methylbutyrate, Fenchol,

Fernlol<sup>TM</sup>, Florilys<sup>TM</sup>, Geraniol, Geranyl Acetate, Geranyl Nitrile, Glidmint<sup>TM</sup> Mint oils, Glidox<sup>TM</sup>, Grapefruit oils, trans-2-Hexenal, trans-2-Hexenol, cis-3-Hexenyl Isovalerate, cis-3-Hexanyl-2-methylbutyrate, Hexyl Isovalerate, Hexyl-2-methylbutyrate, Hydroxycitronellal, Ionone, Isobornyl Methylether, Linalool, Linalool Oxide, Linalyl  
5 Acetate, Menthane Hydroperoxide, l-Methyl Acetate, Methyl Hexyl Ether, Methyl-2-methylbutyrate, 2-Methylbutyl Isovalerate, Myrcene, Nerol, Neryl Acetate, 3-Octanol, 3-Octyl Acetate, Phenyl Ethyl-2-methylbutyrate, Petitgrain oil, cis-Pinane, Pinane Hydroperoxide, Pinanol, Pine Ester, Pine Needle oils, Pine oil, alpha-Pinene, beta-Pinene, alpha-Pinene Oxide, Plinol, Plinyl Acetate, Pseudo Ionone, Rhodinol, Rhodiny  
10 Acetate, Spice oils, alpha-Terpinene, gamma-Terpinene, Terpinene-4-OL, Terpeneol, Terpinolene, Terpinyl Acetate, Tetrahydrolinalool, Tetrahydrolinalyl Acetate, Tetrahydromyrcenol, Tetralol<sup>®</sup>, Tomato oils, Vitalizair, Zestoral<sup>TM</sup>, Hinokitiol<sup>TM</sup> and Thujopsis Dolabrata<sup>TM</sup>.

One of the nonionic surfactants used in the instant composition is a higher  
15 aliphatic, primary alcohol containing about 9-15 carbon atoms, preferably a C<sub>9</sub>-C<sub>11</sub> alkanol condensed with 2 to 3 moles of ethylene oxide.

The other nonionic surfactant used in the instant composition is a higher aliphatic primary alcohol containing 9 to 15 carbon atoms, preferably a C<sub>9</sub>-C<sub>11</sub> alkanol condensed with about 6 to about 9 moles, more preferably about 7 to about 8.5 moles  
20 of ethylene oxide.

The blend of cosurfactants consists of a C<sub>1</sub>-C<sub>5</sub> alkyl ether of mono, di or triethylene glycol and a C<sub>1</sub>-C<sub>5</sub> alkyl ether of mono, di or tripropylene glycol.

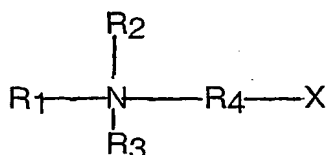
Satisfactory glycol ethers are ethylene glycol monobutyl ether (butyl cellosolve), diethylene glycol monobutyl ether (butyl carbitol), triethylene glycol monobutyl ether,  
25 mono, di, tri propylene glycol monobutyl ether, mono, di, tripropylene glycol monomethyl ether, propylene glycol monomethyl ether, ethylene glycol monohexyl ether, diethylene glycol monohexyl ether, propylene glycol tertiary butyl ether, ethylene glycol monoethyl ether, ethylene glycol monomethyl ether, ethylene glycol monopropyl



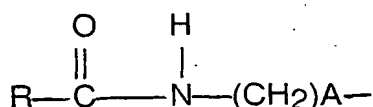
ether, ethylene glycol monopentyl ether, diethylene glycol monomethyl ether, diethylene glycol monoethyl ether, diethylene glycol monopropyl ether, diethylene glycol monopentyl ether, triethylene glycol monomethyl ether, triethylene glycol monoethyl ether, triethylene glycol monopropyl ether, triethylene glycol monopentyl ether, triethylene glycol monohexyl ether, mono, di, tripropylene glycol monoethyl ether, mono, di tripropylene glycol monopropyl ether, mono, di, tripropylene glycol monopentyl ether, mono, di, tripropylene glycol monohexyl ether, mono, di, tributylene glycol monomethyl ether, mono, di, tributylene glycol monoethyl ether, mono, di, tributylene glycol monopropyl ether, mono, di, tributylene glycol monobutyl ether, mono, di, tributylene glycol monopentyl ether and mono, di, tributylene glycol monohexyl ether, ethylene glycol monoacetate and dipropylene glycol propionate.

Water soluble solvent useful in the instant compositions are C<sub>1</sub>-C<sub>4</sub> alcohols such as methanol, ethanol and isopropanol which can be used in blend with the aforementioned glycol ethers blends weight, wherein the ratios of glycol ethers and alcohol are 1:5 to 5:1, more preferably 1:1.

The instant composition contains 0.1% to 10%, more preferably 0.2% to 5%, by weight of an amphoteric surfactant. The amphoteric surfactant can be a water-soluble betaine having the general formula:



wherein x<sup>-</sup> is selected from the group consisting of CO<sub>2</sub><sup>-</sup> and SO<sub>3</sub><sup>-</sup> and R<sub>1</sub> is an alkyl group having 10 to about 20 carbon atoms, preferably 12 to 16 carbon atoms, or an amido radical:



wherein R is an alkyl group having about 9 to 19 carbon atoms and a is the integer 1 to 4; R<sub>2</sub> and R<sub>3</sub> are each alkyl groups having 1 to 3 carbon atoms and preferably 1

carbon; R<sub>4</sub> is an alkylene or hydroxyalkylene group having from 1 to 4 carbon atoms and, optionally, one hydroxyl group. typical alkyldimethyl betaines include decyl dimethyl betaine or 2-(N-decyl-N, N-dimethyl-ammonia) acetate, coco dimethyl betaine or 2-(N-coco N, N-dimethylammonia) acetate, myristyl dimethyl betaine, plamityl dimethyl betaine, lauryl dimethyl betaine, cetyl dimethyl betaine, stearyl dimethyl betaine, etc. The amido betaines similarly include cocoamidoethylbetaine, cocoamidopropyl betaine and the like. A preferred betaine is coco (C<sub>8</sub>-C<sub>18</sub>) amidopropyl dimethyl betaine. Two preferred betaine surfactants are Rewoteric AMB 13 and Golmschmidt betaine L7.

The preferred anionic surfactant used in the instant composition is a paraffin sulfonates containing 10 to 20, preferably 13 to 17 carbon atoms. Primary paraffin sulfonates are made by reacting long-chain alpha olefins and bisulfites and paraffin sulfonates having the sulfonate group distributed along the paraffin chain are shown in U.S. Patent Nos. 2,503,280; 2,507,088; 3,260,744; 3,372,188 and German Patent 735,096.

The pH of the composition will be adjusted to pH 2.7 to 4.5, preferably 3 to 4 with NaOH, KOH, but preferably with an organic alkalinity donor which will not leave streaks such as diethanolamine or triethanolamine.

The final essential ingredient in the inventive compositions having improved interfacial tension properties is water. The proportion of water in the compositions generally is in the range of 20% to 97%, preferably 70% to 97% by weight.

In addition to the above-described essential ingredients, the compositions of this invention may often and preferably do contain one or more additional ingredients which serve to improve overall product performance.

The multi purpose liquid cleaning composition of this invention may, if desired, also contain other components either to provide additional effect or to make the product more attractive to the consumer. The following are mentioned by way of example: Colors or dyes in amounts up to 0.5% by weight, 2,6-di-tert.butyl-p-cresol, etc., in

amounts up to 2% by weight; and pH adjusting agents, such as sulfuric acid or sodium hydroxide, as needed. Furthermore, if opaque compositions are desired, up to 4% by weight of an opacifier may be added.

5 In their final form, the multi purpose liquids are clear compositions and exhibit stability at reduced and increased temperatures. More specifically, such compositions remain clear and stable in the range of 5°C to 50°C, especially 10°C to 43°C.

The compositions are directly ready for use as desired and only minimal rinsing is required and substantially no residue or streaks are left behind. Furthermore, because the compositions are free of detergent builders such as alkali metal  
10 polyphosphates they are environmentally acceptable and provide a better "shine" on cleaned hard surfaces.

When intended for use in the neat form, the liquid compositions can be packaged in a pump-type sprayer for the so-called spray-and-wipe type of application. The composition can also be dispensed from a non woven or fabric towel which can be  
15 used once and discarded or reused several times with adequate rinsing between usage.

Because the compositions as prepared are aqueous liquid formulations , the compositions are easily prepared simply by combining all the ingredients in a suitable vessel or container. The order of mixing the ingredients is not particularly important  
20 and generally the various ingredients can be added sequentially or all at once or in the form of aqueous solutions of each or all of the primary detergents and cosurfactants can be separately prepared and combined with each other and with the perfume. It is not necessary to use elevated temperatures in the formation step and room temperature is sufficient.

25 The instant formulas explicitly exclude alkali metal silicates and alkali metal builders such as alkali metal polyphosphates, alkali metal carbonates, alkali metal phosphonates and alkali metal citrates because these materials, if used in the instant composition, would cause the composition to have a high pH as well as leaving residue on the surface being cleaned.

The following examples illustrate liquid cleaning compositions of the described invention. Unless otherwise specified, all percentages are by weight. The exemplified compositions are illustrative only and do not limit the scope of the invention. Unless otherwise specified, the proportions in the examples and elsewhere in the specification are by weight.

### Example 1

The following compositions in wt. % were prepared by simple mixing procedure:

	1	2	3
Lactic acid	1.5	1	1.5
C9-C11 alcohol EO 2.5:1 nonionic surfactant	0.2	0.2	0.2
C13-17 Parafin sulfonate	1.2	1.2	0.6
Diethylene glycol N-butyl ether	2	2	2
Cocoamido propyl dimethyl betaine	0.45	0.45	0.45
Propylene glycol n-butyl ether	2	2	2
Ethanol	2	2	2
Triethanol amine	0.8	0.57	1.0
Perfume	0.4	0.4	0.4
Water	Bal.	Bal.	Bal.
Ph	3.5	3.5	3.5
Disinfecting results neat EN 1276 Staphylococcus aureus	pass	pass	pass
degreasing test neat	good	good	good
Residue	good	good	good
oil uptake oil level /surfactant level	1.42		

The test protocol used to assess disinfection is the well known European EN 1276 test which uses a strain of bacteria, a short contact time and calls for a bacteria count reduction of 5 log.

In summary, the described invention broadly relates to an improved microemulsion composition containing an anionic surfactant, a nonionic surfactant and an amphoteric surfactant, a water soluble solvent, a hydrocarbon ingredient, a proton donating agent and water.

WHAT IS CLAIMED:

1. A microemulsion composition comprising approximately by weight:
  - (a) 0.1% to 5% of a proton donating agent,
  - (b) 0 to 4% of at least one disinfecting agent;
  - 5 (c) 0 to 10% of an ethoxylated nonionic surfactant formed from a C<sub>9</sub>-C<sub>11</sub> alkanol and about 6 to about 9 moles of ethylene oxide;
  - (d) 0.1% to about 4.0% of an ethoxylated nonionic surfactant formed from a C<sub>9</sub>-C<sub>11</sub> alkanol and about 2 to about 3 moles of ethylene oxide;
  - (e) 0.1% to 10% of an amphoteric surfactant;
  - 10 (f) 1.0% to 10% of at least two different glycol ethers; and
  - (g) the balance being water.
2. The composition according to Claim 1, further including a C<sub>1</sub>-C<sub>4</sub> alkanol.
3. The composition according to Claim 2, wherein one of said glycol ether is propylene glycol N-butyl ether.
- 15 4. The composition according to Claim 1, wherein said proton donating agent is a hydroxy containing organic acid.
5. The composition according to Claim 1, further including an anionic surfactant.

## INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 01/10688

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C11D17/00 C11D3/20 C11D3/00 C11D1/94 C11D3/48

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	US 6 121 224 A (DORMAL DIDIER ET AL) 19 September 2000 (2000-09-19) claims 1-7; example 1	1-4
A	US 5 294 364 A (THOMAS MICHEL ET AL) 15 March 1994 (1994-03-15) column 2, line 43 - column 3, line 61 column 7, line 8-50 claims 1-13; examples	1, 2, 4, 5
A	EP 0 842 606 A (PROCTER & GAMBLE) 20 May 1998 (1998-05-20) claims 1-9; examples I-VI	1-3
A	US 6 046 151 A (DRAPIER JULIEN ET AL) 4 April 2000 (2000-04-04) claims; examples	1-3, 5

☐ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

## \* Special categories of cited documents:

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- \*Z\* document member of the same patent family

Date of the actual completion of the international search

7 August 2001

Date of mailing of the international search report

16/08/2001

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Bertran Nadal, J

## INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 01/10688

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 6121224 A	19-09-2000	US 5911915 A AU 1815099 A WO 9931216 A	15-06-1999 05-07-1999 24-06-1999
US 5294364 A	15-03-1994	US 5192460 A AT 122383 T AU 2987489 A BR 8900594 A CA 1332338 A DE 68922522 D DE 68922522 T DK 65289 A EP 0336878 A MX 170213 B NO 174430 B PT 89679 A,B US 5039441 A AU 628190 B AU 5892590 A BR 9003715 A CA 2022208 A DD 296697 A EP 0411708 A FI 95926 B GR 90100578 A,B HU 55827 A,B IE 902748 A MX 173185 B NZ 234513 A PT 94852 A TR 26026 A	09-03-1993 15-05-1995 10-08-1989 10-10-1989 11-10-1994 14-06-1995 14-09-1995 11-08-1989 11-10-1989 11-08-1993 24-01-1994 04-10-1989 13-08-1991 10-09-1992 31-01-1991 03-09-1991 01-02-1991 12-12-1991 06-02-1991 29-12-1995 10-12-1991 28-06-1991 27-02-1991 07-02-1994 24-03-1997 14-08-1991 01-11-1993
EP 0842606 A	20-05-1998	AT 190469 T AU 5252298 A BR 9712949 A CN 1244893 A DE 69607178 D DE 69607178 T DK 842606 T ES 2143172 T GR 3032827 T NO 992267 A PT 842606 T TR 9901036 T WO 9821307 A US 6114298 A	15-04-2000 03-06-1998 20-06-2000 16-02-2000 20-04-2000 14-12-2000 05-06-2000 01-05-2000 30-06-2000 12-07-1999 31-08-2000 21-07-1999 22-05-1998 05-09-2000
US 6046151 A	04-04-2000	US 5874393 A US 5580848 A US 5529723 A AU 6884698 A EP 0975733 A WO 9846721 A US 6030935 A US 6121228 A US 6048834 A US 5912223 A US 6008180 A	23-02-1999 03-12-1996 25-06-1996 11-11-1998 02-02-2000 22-10-1998 29-02-2000 19-09-2000 11-04-2000 15-06-1999 28-12-1999

# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/US 01/10688

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 6046151 A		US 5840676 A	24-11-1998
		AU 699888 B	17-12-1998
		AU 4376496 A	03-07-1996
		CA 2207683 A	20-06-1996
		EP 0797657 A	01-10-1997
		PL 321067 A	24-11-1997
		WO 9618717 A	20-06-1996
		ZA 9510413 A	09-06-1997